

RESERVE COPY  
PATENT SPECIFICATION

1133210



DRAWINGS ATTACHED

1,133,210

Date of Application and filing Complete Specification: 30 Dec., 1965.

No. 55244/65.

Application made in Germany (No. D46163 IVa/45 I) on 30 Dec., 1964.

Complete Specification Published: 13 Nov., 1968.

© Crown Copyright 1968.

Index at acceptance:—C1 A(H11L, H12A, H12X); A5 E(1C7A, 1C15); C3 R(27K1C6B, 27K1C6X, 27K1C8P, 27K1C8R, 27K1C11, 27K1C12, 27K1C14A, 27K1C14B, 27K1C16, 27K1C21, 27K1C26, 27K1C28X, 27K1C29)

Int. Cl.:—C 01 b 33/14

COMPLETE SPECIFICATION

Pest Control Agent.

We, DEUTSCHE GOLD-UND SILBER-SCHEIDESTALT VORMALS ROESSLER, a body corporate organised under the laws of Germany of 9 Weissfrauenstrasse, Frankfurt/Main, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to an agent for controlling animal pests such as, termites, ants, cockroaches, fleas, lice, bugs, flies, mosquitoes, as well as ticks (red spider) and, in particular, the red fowl pest (*dermanyssus gallinae*) found on chicken farms. The pest-control agent according to the invention is easy to use and is harmless both to human beings and to animals.

20 Examples of the most well known pest-control agents are, dichlorodiphenyl-trichloromethylethane, hexachlorocyclohexane, phosphoric acid esters and methane sulphonic acid fluoride, which are used in the form of powders, solutions and gases.

25 Insecticides such as these kill off parasites by contact. Alternatively, they may be used as a feed poison.

30 All contact insecticides are poisons whose use has to be accompanied by special precautionary measures in view of their toxicity, even to human beings and domestic animals. In addition, a certain resistance is often developed against them. Further, they do not possess any ovicidal activity because eggshells consist of substances which do not have any affinity for the contact insecticides or their active ingredients.

35 The insecticides can be used in many ways, depending upon the type of parasite to be controlled. To control *dermanyssus gallinae*, the insecticide is applied by brushing on to the perches, in the corners of batteries, to nests and to any cracks present in the batteries. The disadvantage of using the insecticide in this

way is that it is extremely difficult to handle because this method of application is only practicable for stalls, etc. intended to accommodate small animals. It would be too expensive and laborious to use on chicken farms.

45 Another method of application comprises scattering the preparations in powder form on to the animals and/or bathing them in a solution of the insecticide. This method of application, too, is complicated and is even impossible to use on a large scale. Similarly, the atomisation and spraying of liquid preparations is only applicable to a limited extent because the accommodation to be treated has to be cleared of both human beings and animals before and during the period of activity of the insecticide. Nevertheless, this method of application is the most common. Since the exact location of the parasites is always known, it is not difficult to spray them with the atomised liquid. On the other hand, all the products currently on the market have to be very carefully handled because there is a danger of poisoning both for human beings and for animals. The animals must in any case be removed from the stall or the box and as a result, they naturally become nervous, and experience has shown that, in the case of hens, nervous tension affects egg output. Apart from this, the method is also time-consuming.

50 55 60 65 70 75 80 85  
Yet another method of application comprises treating stalls with so-called fumigants. The disadvantage of such procedure is that the animals can only be left in their stalls for a specific period. If, in this case, the instructions for use are not carefully followed, there is a serious danger of poisoning. In addition, fumigation can only be effectively carried out if the stall is completely sealed. Doors, windows, cracks and ventilator openings must be closed so that the air is unable to enter the stall. Following fumigation, all the animals have to be led out of their stalls in order that the stalls may be ventilated. In addition, these

agents do not exhibit any ovicidal activity and, for this reason, often have to be repeatedly applied.

It is also known that silicas can be used as carriers or even as agents for controlling parasites. The use of quartz powders is forbidden, as specified in 'Nachrichtenblatt fur den deutschen Pflantenschutzdienst', Vol. 21, No. 6, 1941. Other known silica preparations include fluorine-impregnated silicas in powder form silicas in the form of carriers or free-running agents for e.g. DDT in powder form and powdered  $\text{SiO}_2$ .

According to the literature, the effectiveness of silica powders is attributable to their adsorption by the wax cuticles of the parasites which are thus deprived of their body moisture or liquid on which their survival depends. More recent investigations, however, have cast some doubts on this theory, because tests have shown that the parasites are killed not only by hydrophilic fillers, but also by hydrophobic substances.

The advantages of using  $\text{SiO}_2$  in these known forms are embodied in their physical effectiveness, nontoxicity and in the absence of development of resistance. They are, however, attended by some disadvantages such as their sensitivity to atmospheric moisture which weakens their activity, and their weak adhesion to the articles to be treated for example, perches and walls. They are also difficult to apply because the powder cannot be satisfactorily localised. In addition,  $\text{SiO}_2$  can hardly be used in the open due to atmospheric moisture, mists, rain and wind and its use in enclosed spaces (chicken farms) is also unfavourable because the animals are affected by the continuous, fine  $\text{SiO}_2$ -‘rain’ as a result of which their egg output is reduced. Further,  $\text{SiO}_2$ -powder would not effect all the parasites because, by day, they withdraw to areas which cannot be reached by the powder. Neither has silica powder any ovicidal activity. For this reason, it has to be repeatedly applied in order to destroy the succeeding generations.

The disadvantages referred to above attending the application of highly dispersed silicas in powder form, prompted the carrying out of some tests with aqueous dispersions of highly dispersed silicas prepared by methods known *per se*.

These tests were carried out with standard silica dispersions containing, respectively, 140, 150, 280 and 400 g. of  $\text{SiO}_2$  per 1000 ml. The dispersions were applied with the atomisers normally used in pest control. Unfortunately, spraying of the aqueous silica dispersions had to be stopped after only a very short time because the nozzles of the atomisers were constantly obstructed. Repeated thorough cleaning of the nozzles was not sufficient to overcome this drawback. Apart from this considerable disadvantage attending the practical application of the aqueous silica dispersions,

it was found that the dispersions themselves possessed neither adequate structural viscosity nor adequate strength of adhesion. As a result, it is impossible in cases where these aqueous silica dispersions are applied by spraying, to obtain a uniform adhesive layer, particularly on sloping and vertical surfaces. The addition of wetting agents, emulsifiers, and substances affecting viscosity to the dispersions during their preparation, would not result in the formation of suitable dispersions free from the aforementioned disadvantages because additives such as these which must be present in ordinary silica dispersions used as pest-control agents coagulate the dispersions and detrimentally affect their stability.

The object of this invention is to provide a pest-control agent based on an aqueous silica dispersion which, on the one hand, is harmless both to human beings and animals and, on the other hand, adheres well following its application and in addition is not attended by the disadvantages of the known agents.

The agent provided by the invention consists of an aqueous dispersion of finely divided silica, an aqueous polyethylene imine paste and a wetting agent or emulsifier. Preferably a substance which imparts structural viscosity and/or a substance which imparts consistency are added to the Agent.

As used herein the term “Substance which imparts structural viscosity” means a substance which imparts thisotropic properties to the agent giving it sufficient fluency during spraying but sufficient viscosity when in contact with the object being sprayed to prevent the agent from running off, and the term “substance which imparts consistency” means a substance which produces a more intensive wetting of the ectoparasite by virtue of its protective colloid properties.

It has surprisingly been found that aqueous silica dispersions prepared with the aid of polyethylene imine, exhibit outstanding strength of adhesion and, for this reason, are particularly suitable for controlling *dermanyssus gallinae* on chicken farms.

Dispersions for use in the agent according to the invention may be prepared by thoroughly dissolving aqueous 50% by weight polyethylene imine (PEI) paste in distilled water contained in an agitation vessel, and by subsequently introducing ultra-finely divided silica, preferably silica aerogel, into this solution with stirring, and dispersing it by means of high speed stirrers, particularly suitable for this purpose.

The finely-divided silica for use in the present invention is preferably pyrogenic silica aerogel. The silica aerogels do not necessarily have to be pure and may contain extraneous oxides in a quantity of up to 1% by weight. Favourable results can also be obtained with dispersions based on salts of polyethylene imine obtained by reaction with inorganic and

70

75

80

85

90

95

100

105

110

115

120

125

organic acids, preferably phosphoric acid and acetic acid.

Dispersions such as those based on a PEI-salt can be obtained by thoroughly dissolving an aqueous 50% by weight PEI-paste in distilled water contained in an agitation vessel, and by gradually adding 40% by weight acetic acid or formic acid or an inorganic acid such as, for example, phosphoric acid, to the resulting solution with stirring. A finely-divided silica aerogel, preferably with a BET-surface of about 65m<sup>2</sup>/g, is then introduced into the solution and dispersed in it with a high-speed stirrer particularly suitable for this purpose.

Silica aerogels which do not necessarily have to be pure and may even contain extraneous oxides in a quantity of up to 1% by weight are also particularly suitable for the preparation of this type of dispersion.

The properties of the dispersions so important as far as their application is concerned, may be considerably improved by the addition to the dispersions of wetting agents or emulsifiers, substances that impart structural viscosity and substances that impart consistency.

Suitable wetting agents and emulsifiers include polyglycol ethers such as fatty alcohol polyglycol ethers (alkyl polyglycol ethers), alkylarylpolyglycol ethers and polyglycol ethers of fatty acid ethanolamides. Ethoxylated fatty amines are also suitable. In addition quaternary ammonium compounds, for example lauryl pyridinium chloride or lauryl pyridinium bisulphite may also be used as wetting agent or emulsifier. Suitable as the substances which impart consistency are fatty acid alkanolamines.

The substances which impart consistency and the wetting agents and emulsifiers produce an effective "enveloping" and wetting of the ecto parasites, which the PEI—SiO<sub>2</sub> — dispersion cannot do on its own. Although the wetting agents in conjunction with the dispersion cover the surface of the parasites to an extend which is generally sufficient for less serious cases, the bond strength of the SiO<sub>2</sub>-dispersion is considerably improved by the consistency imparting substance in the sense of envelopment, particularly in cases of persistent and heavy attack.

Since the starting SiO<sub>2</sub>-PEI dispersion is thinly liquid and, if sprayed in large quantities on to a wall or on to the perches of a chicken run in the event of heavy attack, would flow away to a greater or lesser extent, the substances imparting structural viscosity are added to this dispersion, firstly to increase its viscosity and secondly to ensure its fluidity during spraying under the mechanical effect of the pressure applied. Accordingly, it is readily sprayed and, on coming into contact with the objects to be treated, undergoes an increase in viscosity and bonds firmly. Thus, the structure of the viscosity is in temporary collapse but is restored again immediately after application.

Suitable substances imparting structural viscosity are products derived from polyvinyl alcohols such as polyvinyl acetate particularly in the form of an emulsion and cellulose derivatives such as carboxymethylcellulose.

Accordingly, the co-operation of all the additives with the PEI-SiO<sub>2</sub> dispersion guarantees an outstanding pesticidal activity, particularly in persistent cases.

If it is required to obtain some indication of colour in the treatment of runs for example, pigments such as titanium dioxide, aluminium silicate, and dyes may also be added to the dispersions.

The dispersions according to the invention may be satisfactorily sprayed or atomised with the atomisers and spray attachments normally used for this purpose, without any danger of the nozzles being obstructed, even after prolonged periods of use. Further, the coating formed following application of the dispersion, even adheres to sloping and vertical surfaces and as a result is equally effective on such surface because there is no danger of running. Practical tests with the dispersions according to the invention, for example to determine their effectiveness in controlling ectoparasites on poultry, particularly *dermanyssus gallinae*, produced satisfactory long-term results, even in cases where the batteries were completely infected. The animals did not have to be removed when the dispersions were sprayed so that they remained undisturbed, and accordingly did not undergo any decrease in egg output. These tests also demonstrated the complete harmlessness of the dispersions to human beings and animals.

The preparation of an agent according to the invention is described in detail in the following Examples:

**EXAMPLE 1**

4 kg. of a 50% by weight aqueous polyethylene imine paste were stirred into and thoroughly dissolved in 83 litres of distilled water. 12 kg. of finely-divided silica aerogel are then stirred into and dispersed in the resulting solution by means of a turbine stirrer. Finally 0.5 kg. of a wetting agent based on an alkylaryl-polyglycol ether are then added to the dispersion. A low-viscosity dispersion with an outstanding strength of adhesion is obtained.

**EXAMPLE 2**

As in Example 1, 2.0 kg. of a 50% by weight aqueous PEI-paste are dissolved in 81.5 litres of distilled water. 1000 ml. of a 40% by weight acetic acid are then gradually run into the resulting solution with stirring. 15.0 kg. of finely-divided silica aerogel are then stirred into this solution. The procedure is then as in Example 1. Finally, 0.5 kg. of alkylaryl-polyglycolether are added as wetting agent. As in Example 1, a low-viscosity dis-

persion with an outstanding strength of adhesion is obtained.

**EXAMPLE 3**

5      20 kg. of a 10% polyvinyl alcohol solution and 1 kg. of a wetting agent, for example based on an alkyl-aryl-polyglycol ether, are added to an aqueous silica dispersion prepared as described in Example 2.

10     A low-viscosity dispersion is obtained which exhibits an outstanding strength of adhesion, coupled with good penetration, particularly into wood and porous walls.

15     Finely-divided precipitated silica, for example silica precipitated from an aqueous alkaline metal silicate solution is also suitable for preparing the agent according to the invention, although in this case, the content of polyethylene imine has to be increased.

20     The results obtained after a single treatment with the agent prepared in example 3 are given in the Table. The numbers refer to the individual batteries on the chicken farm which were treated in the order indicated. For the sake of clarity, the battery arrangement is dia-

grammatically illustrated in the accompanying drawings, in which Fig. 1 illustrates the positions of the individual batteries, whilst Fig. 2 shows a battery on its own. In Fig. 2, 1 is a water trough, 2 the 'scratching' compartment, 3 the feed-carrying conveyor belt, 4 the perches and 5 the laying boxes.

In connection with the Table and drawing, it is pointed out that the tests in question were carried out on a large scale. A run of the kind shown in Figs. 1 and 2 was sprayed with an agent according to the invention. After treatment most of the boxes were completely free of parasites and although some were still affected, the infection was not serious. It must be assumed that, in these tests, the spray did not reach every corner. Alternatively, the extent to which these boxes were affected could have been so serious that not all the parasites were caught. A second treatment was sufficient to kill them all.

35     Battery of a poultry farm after a single treatment with the product according to the invention against *dermanyssus gallinae*.

25

30

35

40

45

**CHECKS**

No.	I		II		III	
	Battery	nest	Battery	nest	Battery	nest
15	-	-	-	-	-	-
14	+	-	+	-	-	-
13	-	-	-	-	-	-
12	-	-	-	-	-	-
11	-	-	-	-	-	-
10	-	-	+	-	-	-
9	-	-	-	-	-	-
8	-	-	-	-	-	-
7	-	+	+	-	++	-
6	-	+	-	+	-	+
5	-	+	-	++	-	+
4	-	+++	-	+++	-	+++
3	-	++	-	+	-	++
2	-	+	-	+	-	+
1	-	+	-	-	-	+

## CHECKS

No.	I		II		III	
	Battery	nest	Battery	nest	Battery	nest
16	-	-	-	-	-	-
17	-	-	-	-	-	-
18	-	-	-	-	-	-
19	-	-	-	-	-	-
20	-	-	-	-	-	-
21	-	-	-	-	+	-
22	+	+	+	-	+	-
23	+	-	+	+	+	+
24	+	-	++	++	++	++
25	+	+	-	+	+	+
26	+	-	+	-	+	-
27	+	+	+	+	+	+
28	-	-	-	-	-	-
29	+++	++	-	-	-	-
30	+	-	-	-	-	-

Meaning of signs

- = no parasites
- + = slight infestation
- ++ = average infestation
- +++ = serious infestation

Temperatures measured in the chicken house on the days of the checks are as follows:—

Check I = 28 °C.  
 Check II = 30 °C.  
 Check III = 31 °C.

**WHAT WE CLAIM IS:—**

1. A pest-control agent comprising an aqueous dispersion of finely-divided silica, an aqueous polyethylene imine paste and a wetting agent or emulsifier. 25

5 2. An agent as claimed in claim 1, comprising in addition a substance which imparts structural viscosity. 30

10 3. An agent as claimed in claim 1 or 2, comprising in addition a substance which imparts consistency. 35

4. An agent as claimed in any of claims 1 to 3, wherein the silica is a finely-divided silica aerogel.

15 5. An agent as claimed in claim 4, wherein the silica aerogel contains up to 1% of an oxide other than silica.

20 6. An agent as claimed in any of claims 1 to 5, wherein the wetting agent or emulsifier is a polyglycol ether, an ethoxylated fatty amine or a quaternary ammonium compound.

7. An agent as claimed in any of claims 2 to 6, wherein the substance which imparts

structural viscosity is a product derived from polyvinyl alcohol or is a cellulose derivative. 25

8. An agent as claimed in any of claims 3 to 6, wherein the substance which imparts consistency is a fatty acid alkanolamine.

9. An agent as claimed in any of claims 1 to 8, including a pigment and/or a dye. 30

10. An agent as claimed in claim 8, including titanium dioxide and/or aluminium silicate as pigment.

11. An agent as claimed in claim 1, substantially as hereinbefore described with reference to any of the examples.

12. A process for combating animal pests utilising an agent as claimed in any of claims 1 to 11. 35

**ELKINGTON AND FIFE,**  
Chartered Patent Agents,  
High Holborn House,  
52/54 High Holborn,  
London, W.C.1.  
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1968.  
Published by the Patent Office, 25 Southampton Buildings, London, W.C.2, from which  
copies may be obtained.

1,133,210

1 SHEET

COMPLETE SPECIFICATION

This drawing is a reproduction of  
the Original on a reduced scale.

FIG. 1.

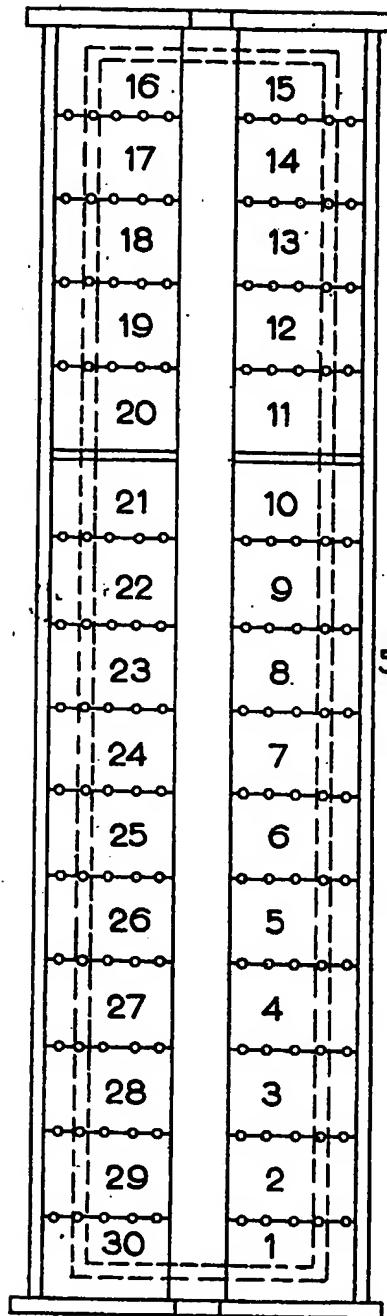


FIG. 2.

